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MELTING PHENOMENON AND LASER ANNEALING IN SEMICONDUCTORS

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Melting Phenomenon and Laser Annealing in Semiconductors*

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Abstract. The work on annealing of displacement damage, dissolution of boron precipitates, and the broadening of dopant profiles in semiconductors after treating with ruby ($\lambda=0.649 \mu\text{m}$, $\tau=15\text{ns}$, $E=1.0\text{-}2.0 \text{ Jcm}^{-2}$), and dye ($\lambda = 0.485 \mu\text{m}$, $\tau= 9\text{ns}$, $E=0.7\text{-}1.3 \text{ Jcm}^{-2}$) laser pulses is reviewed in order to provide convincing evidence for the melting phenomenon and illustrate the mechanism associated with laser annealing. The nature of the solid-liquid interface and the interface instability during rapid solidification is considered in detail. It is shown that solute concentrations after pulsed laser annealing can far exceed retrograde maxima values. However, there is a critical solute concentration above which a planar solid-liquid interface becomes unstable and breaks into a cellular structure. The solute concentrations and cell sizes associated with this instability are calculated using a perturbation theory, and compared with experimental results.

1. Introduction

High power ruby laser pulses have been used to study the removal of displacement damage, the dissolution of precipitates, and the formation of constitutional supercooling cells, and the results have been interpreted consistently on the basis of a melting model (Wang et al 1978, Narayan et al 1980). The basic assumption in this model is that the energy from the electronic system can be rapidly ($<10^{-9}$ s) transferred to the lattice and this energy is then utilized to heat and melt thin ($< 1 \mu\text{m}$) surface layers of materials. Some authors, notably Khaibullin et al (1978) and Van Vechten et al (1979), have speculated on a plasma model. In this model, the energy transfer from the electronic system to the lattice is delayed ($> 200 \text{ ns}$) through the screening of phonon emission, and these authors envisage that the high concentrations of electrons and holes produced during pulsed laser irradiation may cause annealing via enhanced diffusion of point defects and glide and climb of dislocations. At a still higher plasma density ($\sim 8 \times 10^{21}/\text{cm}^{-3}$), electrons excited into antibonding states are proposed to lead to a weak first-order phase transition, resulting in a softening of the lattice. The crystal would then be in a fluid-like state with energy retained primarily in the electronic system. This energy would then be distributed over a much greater depth as carrier diffusion becomes important. As the plasma density decreases, the material passes back through the phase transition, leading to recrystallization. Recently, Lo and Compaan (1980) have attempted to measure the lattice temperature of

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silicon, during pulsed laser irradiation, by Raman scattering techniques, within 10 ns of a dye laser pulse ($\lambda = 0.485 \mu\text{m}$, $\tau = 9 \text{ ns}$) in the energy range of 0.7 to 1.0 Jcm^{-2} . They reported a temperature rise of 300°C for a 1.0 Jcm^{-2} laser pulse, thus lending support to the plasma annealing model.

In this paper, results on the annealing of displacement damage, on the dissolution of precipitation, and on the formation of constitutional supercooling cells obtained using dye laser pulses are discussed to establish melting as a primary mechanism of laser annealing. These results are similar to those obtained using pulsed ruby laser irradiations. Solute concentrations (in substitutional sites) achieved under laser melt-quenching conditions can far exceed the retrograde maxima values determined from phase diagrams. However, at a given velocity of solidification there is a limiting solute concentration ($C_{s(\text{min})}$) above which the planar solid-liquid interface becomes unstable, and breaks into cellular structures. From the study of cell sizes and the limiting concentrations, we are able to obtain detailed information on rapid solidification.

2. Laser Annealing of Displacement Damage

In this section, results on the annealing of displacement damage in ion implanted silicon specimens, after irradiation with dye laser pulses, are described. Figure 1 shows annealing results in terms of x-section TEM micrographs from arsenic implanted (200 keV $^{75}\text{As}^+$, dose = $1.0 \times 10^{15} \text{ cm}^{-2}$) specimens which contained an amorphous layer $\sim 1800 \text{ \AA}$ thick followed by a band ($\sim 700 \text{ \AA}$ thick) of dislocation loops. After irradiating with a 0.75 Jcm^{-2} laser pulse having Gaussian energy distribution across the spot, the central regions (20-100 μm across) were found to be defect free as shown in Fig. 1(d). This annealed region was surrounded by a narrow region containing V-shaped dislocations [Fig. 1(c)], which in turn was followed by an annular polycrystalline region [Fig. 1(b)]. The average size of polycrystalline grains was smaller in the outer regions [as shown in Fig. 1(a)] where annealing was not affected throughout the amorphous layer. From the comparison of these results with those obtained using uniform ruby laser pulses, the following was concluded. In the central region the pulse energy density was high enough such that the melt-front exceeded the depth of the damaged layer, and the specimens grew defect-free with the same perfection as the underlying substrate. In the region immediately surrounding the central region the melt-front reached the dislocation band, and the loops intersected by the melt-front grew back as two segments of V-shaped dislocations. The dislocations of a given Burgers vector were found to grow in a specific direction, which was characteristic of crystal growth from the melt (Narayan and Young, 1979). In the outer regions where melt-front did not reach the crystalline substrate, polycrystals were observed.

The results on laser annealing of dislocation loops in boron implanted (180 keV, $6.0 \times 10^{15} \text{ cm}^{-2}$) silicon specimens, which were irradiated with a dye-laser pulse of 1.23 Jcm^{-2} , are presented in Fig. 2. The thickness of annealed regions from a x-section image [Fig. 2(a)] of the outer regions of the laser spot, was determined to be 500 $\text{\AA}$. The depth of annealing reached to a maximum value of 4000 $\text{\AA}$ near the center of the laser spot as shown in Fig. 2(b). The as-implanted specimens contained loops to a depth of $\sim 0.60 \mu\text{m}$. The removal of dislocation loops and the sharpness of

0.3 μm

a

b

c

Fig. 1 X-section electron micrographs showing microstructures (for $E = 0.75 \text{ Jcm}^{-2}$ pulse) from outer regions to the middle of the laser spot (as a function of increasing pulse energy density). (a) Polycrystalline layer with underlying amorphous layers; (b) polycrystalline layer with no amorphous layer; (c) V-shaped dislocations growing from the dislocation band below the amorphous layer; and (d) nearly "complete" annealing.

transitions observed between the annealed and unannealed regions in the micrographs, are completely consistent with the melting model.

The arsenic concentration profiles from the central, annealed regions for the 0.75 and 0.92 Jcm^{-2} laser pulses are shown in Figs. 3(a) and 3(b) respectively. The as-implanted profiles are Gaussian as expected. After laser annealing, a significant dopant redistribution occurs both toward the surface and into the deeper regions of the crystal. The drastic changes in the dopant profiles after laser annealing are consistent with melting. The inset in Fig. 3(b) shows the experimentally deduced melt-front penetration vs time, which was constructed from the estimated velocity of solidification ($\sim 6.0 \text{ ms}^{-1}$), total melt-lifetime (60 ns), and the maximum depth of melt-front penetration of $3300 \text{ \AA}$ obtained from dislocation growth experiments. From Fig. 3(b), the average diffusion distance at a depth of $2000 \text{ \AA}$ was estimated to be $250 \text{ \AA}$, and the corresponding melt-lifetime from the inset was 20 ns. Assuming unidirectional diffusion and equating $\sqrt{Dt} = 250 \text{ \AA}$, we obtain the value of D as $3.0 \times 10^{-4} \text{ cm}^2 \text{ s}^{-1}$, which was found to be in good agreement with the value of the diffusion coefficient of arsenic in liquid silicon. Recently, detailed numerical calculations (Narayan and Wang, unpublished) have been performed to fit the entire dopant profile after laser annealing, and essentially the same value of D was extracted from these results.

Fig. 2 TEM x-section images showing the thicknesses of laser-annealed ($E = 1.23 \text{ Jcm}^{-2}$) regions: in boron-implanted (180 keV , $16 \times 10^{15} \text{ cm}^{-2}$) Si specimens: (a) outer regions of the laser spot, (b) center of the laser spot.

3. Dissolution of Precipitates

In this section, we summarize the results on the dissolution of boron precipitates, obtained from specimens irradiated with laser pulses. The precipitates were produced by a conventional solid-state diffusion process at 950°C using B_2O_3 . Figure 4(a) shows these precipitates; the average size of the precipitates was determined to be $100 \text{ \AA}$, and they were confined in the top $300 \text{ \AA}$ thick layer. A dye laser pulse of 1.17 Jcm^{-2} leads to dissolution of these precipitates so that no defects are observed, as shown in Fig. 4(b). The boron concentration profiles before and after laser annealing are shown in Fig. 5. The redistribution or the transport of boron, which was contained in the precipitates near the surface, occurred over a distance of about $1500 \text{ \AA}$. It should be remarked that using known diffusion coefficients, the drastic profile changes observed after laser annealing are consistent with impurity diffusion in the liquid, and these results cannot be explained by diffusion in the solid state. For example, the diffusion coefficient of B in the solid state, even close to the melting point, is about $10^{-12} \text{ cm}^2 \text{ s}^{-1}$ (Grove, 1967). If we take the characteristic time τ of the laser annealing to be approximately ten times the pulse duration (a large overestimate), the diffusion length is still estimated to be about $3 \times 10^{-2} \text{ \AA}$. The presence of a plasma could enhance (via ionization effects) the migration of already present point defects, and could enhance the glide of dislocations; but there is no realistic plasma model to explain the break-up and dissolution of precipitates.

Fig. 3 (a) Arsenic concentration profiles before and after the dye laser annealing: $E = 0.75 \text{ J cm}^{-2}$; (b) The arsenic concentration profile before and after the dye laser annealing: $E = 0.92 \text{ J cm}^{-2}$. The inset shows melt-front penetration as a function of time.

4. Metastable Alloying and the Formation of Constitutional Supercooling Cells

Following the melting model, pulsed laser annealing essentially involves crystal growth with the underlying substrate acting as a seed. Since the estimated velocities of solidification are of the order of meters per second and can be varied by controlling the laser parameters or the substrate temperature, many investigations have been made to determine the solubility limits, and the dependence of distribution coefficient (k') on the velocity (White et al, 1980, Cullis et al, 1980). Substitutional solute concentrations under laser annealing conditions have been found to far exceed the equilibrium solubility limits and even the retrograde maxima by factors exceeding 500. However, at a given velocity there is a limiting value of solute concentrations ($C_{S(\text{min})}$) which can be incorporated substitutionally above this concentration the planar solid-liquid interface is no longer stable and it breaks into cellular structures (Narayan 1980).

In this section, we summarize the results obtained using ruby laser pulses on interface instability and cell formation. Similar results obtained from the dye laser pulses are reported elsewhere (Narayan et al, 1981). Figure 6 shows ion channeling, backscattering results on an indium implanted, laser annealed silicon specimen. The plot shows the substitutional and the total indium concentration as a function of depth. As the

b

0.2 μm

Fig. 4 Dissolution of boron precipitates in laser-annealed silicon: (a) boron precipitates in as-diffused (at 950°C, 30 min) silicon, and (b) precipitates dissolved after laser annealing.

Fig. 5 Boron concentration profile before and after laser annealing ($E = 1.17 \text{ Jcm}^{-2}$).

solid-liquid interface sweeps back to the surface, the concentration profile is modified according to the distribution coefficient (ratio of solubilities in the solid to the liquid at the interface) and diffusion coefficient of indium in liquid silicon. Solute concentration profiles have been calculated, and by fitting these profiles to the observed profiles, the values of distribution coefficients can be obtained. The values of k' extracted from this analysis are shown in Table I. A comparison of these values with the equilibrium values obtained from the phase diagram indicates that at such high velocities k' for As approaches unity, while for other impurities, it is still less than unity. The latter, even at such high velocities, exhibit impurity segregation. The point where the substitutional and total concentrations start departing determines the maximum concentration which can be incorporated at this velocity of solidification. Table II shows solute concentrations for various impurities

Fig. 6 Comparison of total and substitutional concentrations for ^{115}In (125 keV, $1.25 \times 10^{16} \text{ cm}^{-2}$) after laser annealing (after White et al 1980).

Fig. 7 TEM images showing formation of cell structures in indium implanted (125 keV In, $1.0 \times 10^{16} \text{ cm}^{-2}$) laser annealed (ruby laser, $E = 1.6 \text{ Jcm}^{-2}$, $\tau = 15 \text{ ns}$) silicon: (a) Plan view of cells in (100) Si, (b) (100) diffraction pattern, (c) dark-field micrograph using one of the In spots, (d) cells in (111) silicon.

at 4.0 ms^{-1} ; the retrograde maxima are also included in this table. The ratio of observed concentrations to the retrograde maxima vary from a factor of 4 for As to a factor of 500 for Bi in silicon. For arsenic in silicon, the observed concentrations approach the values obtained from T_0 diagrams, corresponding to a diffusionless transformation from liquid to solid, while for others these values are still smaller than the T_0 concentrations (Cahn et al 1980). Baker and Cahn (1969) have presented arguments to show that solute concentrations higher than the retrograde maxima represent positive deviations from the equilibrium, and provide direct evidence for solute trapping and the formation of metastable alloys. Figure 6 also shows a depth at which substitutional concentrations of indium become constant, and the amount of solute corresponding to the difference between the total and the substitutional concentration is incorporated into the cells. It is believed that at this depth the planar solid-liquid interface becomes unstable and the instability results in the formation of cell structures (Narayan 1980).

The electron micrographs in Fig. 7 show constitutional supercooling cells. Figure 7(a) is a plan-view micrograph from a (100) specimen irradiated with a 1.60 Jcm^{-2} ruby laser pulse. The average cell size was determined to be $500 \text{ \AA}$. The interior of each cell was found to be defect-free

Table I. Equilibrium (k_0) and Experimentally Deduced Distribution Coefficients

Dopant	k_0	k'
As	0.3	1
Sb	0.023	0.7
Ga	0.008	0.2
In	0.0004	0.15
Bi	0.0007	0.4

Table II. Solubility Limits

Dopant	Retrograde Maxima (cm^{-3})	Concentrations After Pulsed Laser Annealing (cm^{-3})
As	1.5×10^{21}	6×10^{21}
Sb	7×10^{19}	1.3×10^{21}
Ga	4.5×10^{19}	4.5×10^{20}
In	8×10^{17}	1.5×10^{20}
Bi	8×10^{17}	4×10^{20}

silicon. Figure 7(b) is a selected-area diffraction pattern from the specimen shown in Fig. 7(a), which contains, in addition to 200 and 220 silicon spots, 110 indium diffraction spots, the latter appearing near the forbidden 200 silicon spots. The diffraction results also indicate that indium has grown epitaxially on the silicon substrate. Figure 7(c) is a dark-field micrograph obtained using one of the In spots, and it shows that virtually all the indium has segregated into the cell walls. Under similar laser irradiation conditions, the cells for the $\langle 111 \rangle$ orientation were observed to have a different morphology [Fig. 1(d)] from that for the $\langle 100 \rangle$ orientation. The average cell size was found to increase from 500 Å in the $\langle 100 \rangle$ to 700 Å in the $\langle 111 \rangle$ orientation. The cross-section micrograph in Fig. 8, from a $\langle 100 \rangle$ specimen, showed that the cells started at a depth of 950 Å, with cell walls aligned close to the surface normal. This depth of cell formation is consistent with the ion channeling, backscattering results of Fig. 6. The solute concentration and the cell size associated with morphological instability can be calculated using the perturbation theory originally developed by Mullins and Sekerka (1964). This theory takes into account constitutional supercooling, surface tension, and the evolution of latent heat, including the heat transport from the interface. In their theory, we have incorporated the dependence of the distribution coefficient (k') on the velocity of solidification (Narayan 1980, Wood 1980). Assuming local equilibrium at an isotropic solid-liquid interface, the limiting concentration C'_S is given by

$$C'_S = \frac{D_L k'}{m V \bar{\alpha} (k' - 1)} \left[\frac{k_L G_L}{k_L + k_S} \left(\alpha_L - \frac{V}{k_L} \right) + \frac{k_S G_S}{k_S + k_L} \left(\alpha_S + \frac{V}{k_S} \right) \right] \left[1 + \frac{2 k'}{(1 + (2 D_L \omega / V)^2)^{1/2} - 1} \right], \quad (1)$$

Fig. 8 X-section micrograph from a specimen corresponding to Fig. 7(a).

a

$$\alpha_L = (V/2K_L) + [(V/2K_L)^2 + \omega^2]^{1/2}$$

$$\alpha_S = -(V/2K_S) + [(V/2K_S)^2 + \omega^2]^{1/2}$$

$$\bar{a} = (k_S \alpha_S + k_L \alpha_L) / (k_S + k_L)$$

where D_L is the impurity diffusion coefficient in liquid, V is the velocity of solidification, m is the liquidus slope in the phase diagram, k_L and k_S are the thermal conductivities, and K_L and K_S are the thermal diffusivities, of the liquid and solid, respectively, G_L and G_S are the thermal gradients in the liquid and solid respectively, T_m is the melting point of the planar interface in the absence of solute, Γ is the ratio of the solid-liquid surface energy to the latent heat of fusion per unit volume, ω is the spatial frequency associated with the instability, and $k' = C_S/C_L$ where C_S and C_L are the interface solubilities in the solid and liquid respectively. k' depends upon the velocity of solidification and the following function was used in the calculations (Wood, 1980),

$$k' = k_0 \exp \{-RT \ln k_0\} (1 - \exp \{- (V/V_0)\}) / RT \quad (2)$$

where k_0 is the equilibrium distribution coefficient, and V_0 is a constant which can be determined experimentally.

We have solved $C'_S(\omega)$ as a function of ω for given values of V . The minimum value of $C'_S(\omega)$ gives the stability-instability demarcation. $\lambda_{\min} = 2\pi/\omega_{\min}$ then represents the cell size where $\omega_{\min}$ is the value corresponding to the minimum value of C'_S . The following values of the materials constants were used in the calculations

$$k_L = 70 \text{ Jm}^{-1}\text{s}^{-1}\text{K}^{-1}, k_S = 22 \text{ Jm}^{-1}\text{s}^{-1}\text{K}^{-1}$$

$$D_L = 1.0 \times 10^{-8} \text{ m}^2\text{s}^{-1} \text{ (curve a)} \quad 5.0 \times 10^{-8} \text{ m}^2\text{s}^{-1} \text{ (curve b),}$$

$$k_0 = 4.0 \times 10^{-4}, V_0 = 2.83 \text{ ms}^{-1}$$

$$T_m = 1.3 \times 10^{-7} \text{ K}$$

$$K_L = 3.0 \times 10^{-5} \text{ m}^2\text{s}^{-1}, K_S = 9.4 \times 10^{-6} \text{ m}^2\text{s}^{-1},$$

$$G_S = 6.57 \times 10^8 \text{ K}^{-1}, G_L = 1.33 \times 10^6 \text{ K}^{-1} \text{ (Wood, 1980)}$$

$$m = -400k \text{ (at. fract.)}^{-1}.$$

The results of the calculations of $C'_S(\min)$ and cell size as a function of solidification velocity are displayed in Figs. 9 and 10 respectively. It

Fig. 9 Limiting concentration of instability versus velocity of solidification: curve (a) $D_L = 1.0 \times 10^{-8} \text{ m}^2 \text{ s}^{-1}$, curve (b) $D_L = 5.0 \times 10^{-8} \text{ m}^2 \text{ s}^{-1}$ ($k' = f(v)$ for both curves), and k_0 curve for a fixed of $k' = k_0$ and $D_L = 5.0 \times 10^{-8} \text{ m}^2 \text{ s}^{-1}$.

Fig. 10 Cell size as a function of solidification velocity: a, b, and k_0 curves correspond to those in Fig. 7, and the curve with a constant value of $k' = 0.15$ is included. The value of $k' = 0.15$ was deduced at $V = 4.0 \text{ ms}^{-1}$.

should be pointed out that $C_s^i(\text{min})$, the solute concentration at the onset of instability, is equal to the C_s^{max} , the maximum substitutional solute concentration. The curve (a) is for $D_L = 1.0 \times 10^{-8} \text{ cm}^2 \text{ s}^{-1}$ and the curve (b) is for $D_L = 5.0 \times 10^{-8} \text{ cm}^2 \text{ s}^{-1}$; these values cover the range of D_L given in the literature (Kodera 1965, Shaskhov and Gurevich 1968). The limiting value of solute concentration determined from Fig. 6 is also plotted in Fig. 9, indicating good agreement between the calculations and the experiment. The calculations using the constant value of k_0 , however, give about four orders of magnitude lower value of $C_s(\text{min})$ at $V = 4 \text{ ms}^{-1}$; but, as expected for lower velocities, the two calculations ($k' = f(v)$ and $k' = k_0$) provide the same results. Also plotted in Fig. 9 are limiting concentrations obtained using the classical constitutional supercooling (CS) criterion $MG_C = G_L$, and the modified (CS) criterion $MG_C = (k_L G_L + k_S G_S) / (k_L + k_S)$, where $G_C = VC_S(k' - 1) / D_L k'$. These results indicate the need to use the modified CS criterion, even at lower velocities, for those cases where temperature gradients in the liquid (G_L) and solid (G_S) are vastly different. The agreement between the observed and calculated cell sizes is considered good in view of the uncertainty in laser and materials parameters. From the calculations in Fig. 10, the increase in cell size, from 500 Å for (100) to 700 Å for (111) orientation, suggests a decrease in solidification velocity by ~40% in (111) compared to (100) orientation.

The velocity of solidification can also be varied by changing the substrate temperature. By cooling the substrate to liquid nitrogen temperature, the velocity is increased by 40%, similarly by heating the substrate to 600°C it decreases by 50%. The cell sizes in indium, gallium and bismuth implanted silicon have also been studied as a function of solidification velocity and good agreement with the theory was again obtained (Narayan, unpublished).

Recently we have addressed the effect of surface tension on the cell size. Since the atomic density in {111} planes is higher than in {100} planes, the liquid-solid surface tension in the <111> orientation is expected to be correspondingly higher (by a factor of $\leq 20\%$) than in the <100> orientation (Narayan, unpublished). The calculations show that a change in surface tension of about 20% leads to less than a 5% change in the cell size. Therefore, it is believed that the observed change in cell sizes is primarily due to differences in solidification velocities in the <111> and <100> orientations.

In conclusion, the results (obtained using the dye-laser pulses, the same as those used by Compaan et al) on annealing of displacement damage (amorphous as well as layers containing only dislocation loops) in semiconductors, on the dissolution of boron precipitates, and on the formation of constitutional supercooling cells, can only be interpreted on the basis of a melting model. These results are similar to our earlier results obtained using ruby-laser pulses. The details of melting and associated rapid solidification can be obtained by studying the formation of constitutional supercooling cells and the limiting concentrations of solutes which can be incorporated into the substitutional sites. The calculations of the cell sizes and the limiting concentrations, based upon the perturbation theory of Mullins and Sekerka, are in good agreement with the experimental results when the dependence of the distribution coefficient upon the velocity of solidification is taken into account in the theory.

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